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Behavior of Mechanical Properties In Neutron Irradiated 12Ni-5Cr-3Mo Maraging Steel Plate and Companion Weld Metals

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CONTENTS

ABSTRACT

Changes of Charpy V notch-ductility and tensile strength in neutron-irradiated 12Ni-5Cr-3Mo maraging steel have been evaluated following low (<250°F) and elevated (550 to 740°F) temperature exposure. The study was performed with six heats of 1-in.-thick plate material aged at 900°F for 2 and 20 hr to nominal yield strengths of 160 and 180 ksi, respectively. The long-term thermal stability of both heat-treatment conditions was investigated for the conditions of irradiation. The <250°F and 550°F irradiation performance of matching (12-5-3) and mismatching (17Ni-2Co-3Mo) TIG weld deposits maraged to 180 ksi yield strength was also assessed in this study.

Changes in the general properties of the 12-5-3 maraging steel plate and companion weld metals were found to be rather small with <250°F exposures, indicating good resistance to neutron-induced embrittlement. However, a marked deterioration of notch-ductility properties with long-term exposure at elevated temperature was revealed and traced to a nonnuclear thermal instability. The observed instability is believed to be a continuation of aging processes at temperatures well below the initial maraging temperature. Extended time-at-temperature treatments indicate that service above 550°F may produce sufficient changes in properties for failure by low-energy tear. Aging treatments of 1900 to 3300 hours' duration increased the yield and tensile strengths of the 12-5-3 alloy by as much as 52.0 and 53.3 ksi, respectively, while not altering appreciably the percentage of elongation and the reduction in area.

PROBLEM STATUS

This is an interim report on one phase of the problem; work is continuing.

AUTHORIZATION

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BEHAVIOR OF MECHANICAL PROPERTIES IN NEUTRON IRRADIATED 12Ni-5Cr-3Mo MARAGING STEEL PLATE AND COMPANION WELD METALS

INTRODUCTION

Maraging steels feature yield strength and notch toughness that are attractive for nuclear power applications. Extensive evaluations of nuclear environmental effects on the mechanical properties of other structural steels have also suggested that steels of higher yield strength are less sensitive to neutron radiation embrittlement (1). Thus, a determination of the general response of maraging steels to neutron irradiation is of great interest. Several melts in two heat-treatment conditions and two weld metals of different alloy chemistry were subsequently obtained for radiation assessments of properties' retention.

Screening experiments conducted initially on plate and weld deposits involved low irradiation temperatures (<250°F) and low fluence accumulations (n/cm^2 > 1 MeV). The promising results led to the extension of experimental irradiation conditions to elevated temperature exposures (>500°F) as well as to high fluence accumulations. Simultaneously, thermal conditioning studies were conducted to measure the stability of the basic aging mechanisms for this type steel with long-term exposure (months) at temperatures up to 740°F. This report presents and summarizes the results of these studies including observations on the unexpected thermal response characteristics of the plate material.

MATERIALS

Chemistries of the 1-in. plates and TIG-deposited weld metals of this investigation are given in Table 1. The plates have a nominal 12Ni-5Cr-3Mo composition with minor variations in alloy additions. The weld metals represent matching and mismatched chemistries: 12Ni-5Cr-3Mo and 17Ni-2Co-3Mo. Individual heat-treatment conditions and resulting yield strengths are listed in Table 1. The plates fall into two general categories of yield strength (160 and 180 ksi) according to the aging time (2 and 20 hr). The weld metals were evaluated in the 180-ksi yield-strength condition only.

Preirradiation Charpy V notch-ductility and tension-test data are given in Table 2. Specimens were taken in two layers through the thickness of the 1-in. materials. The Charpy V-notch specimens were oriented in the transverse (WR: weak) plate direction to provide a more conservative analysis of irradiation performance. Charpy-V specimens in weld metal were taken perpendicular to the welding direction. However, the narrow width of the weld joint dictated that all-weld tensile specimens be used. Tensile specimens taken from plate materials were oriented in both longitudinal (RW) and transverse (WR) test directions. For both weld-metal and plate assessments, the temperature corresponding to the development of a 30-ft-lb Charpy-V level was arbitrarily chosen as the index for evaluating the irradiation damage.

A composite of the microstructures of base plate, heat-affected zone, and weld metal is presented in Fig. 1. The general microstructure is identified as low-carbon martensite with a uniform dispersion of titanium carbonitrides.

Table 1
Chemical Composition and Heat Treatment of 12-5-3 Maraging Steel Plates and Companion Weld Metals (TIG)*

Material Code	Heat Treatment†	Yield Strength (0.2% offset) (ksi)	Chemical Analysis (%)											
			C	Mn	Si	P	S	Ni	Cr	Mo	Ti	Al	Co	V
J1	A	179.1	0.003	0.03	0.06	0.003	0.007	12.1	4.83	3.00	0.24	0.24	—	—
J3	B	170.6	0.003	0.03	0.06	0.003	0.007	12.1	4.96	3.10	0.24	0.24	—	—
J4	B	162.8	0.003	0.03	0.06	0.003	0.007	12.1	4.83	3.10	0.24	0.24	—	—
J5	C	176.2	0.003	0.03	0.06	0.003	0.007	12.1	4.83	3.10	0.24	0.21	—	—
J6	C	184.4	0.007	0.04	0.08	0.005	0.007	11.8	5.08	3.30	0.24	0.14	—	—
J7	C	181.5	0.005	0.03	0.06	0.003	0.007	12.1	4.83	3.10	0.24	0.22	—	—
J8	D	162.6	0.005	0.04	0.05	0.005	0.007	11.8	5.16	3.30	0.24	0.13	—	—
12-5-3 Weld	E	179.0	0.001	0.10	0.08	0.002	0.007	11.9	4.97	3.20	0.24	0.14	0.007	0.02
17-2-3 Weld	E	178.8	0.003	0.05	0.04	0.007	0.007	14.5	1.93	3.10	0.24	0.09	1.45	0.01

*As-deposited, weld-metal composition.
†A — Solution annealed at 1500°F for 2 hr; water quenched; aged at 900°F for 20 hr; water quenched.
B — Solution annealed at 1500°F for 2 hr; water quenched; aged at 900°F for 2 hr; water quenched.
C — Solution annealed at 1500°F for 1 hr; water quenched; aged at 900°F for 20 hr; water quenched.
D — Solution annealed at 1500°F for 1 hr; water quenched; aged at 900°F for 2 hr; water quenched.
E — Postweld heat treatment; aged at 850°F for 12 hr.

Table 2
Mechanical Properties of 12-5-3 Maraging Steel Plates and Companion Weld Metals

Material Code	Charpy-V 30-ft-lb* Transition Temp. (°F)	Yield Strength† (0.2% offset) (ksi)	Tensile Strength (ksi)	Elongation (1-in. gage) length) (%)	Reduction of Area (%)
J1	-180	179.1	185.1	15.5	64.5
J3	-200	170.6	175.6	16.0	66.0
J4	-160	162.8	170.9	16.6	64.4
J5	-165	176.2	185.3	13.5	58.3
J6	-175	184.4	190.0	13.5	55.7
J7	-180	181.5	188.5	14.5	62.3
J8	-200	162.6	170.6	15.8	64.1
12-5-3 Weld	-70	179.0	188.0	16.0	62.5
17-2-3 Weld	-175	178.8	187.5	16.0	64.7

*Plate: Transverse to primary rolling direction.
Weld: Transverse to welding direction.
†Plate: Transverse to primary rolling direction.
Weld: All weld-metal, 0.252-in.-diameter specimens.

IRRADIATIONS

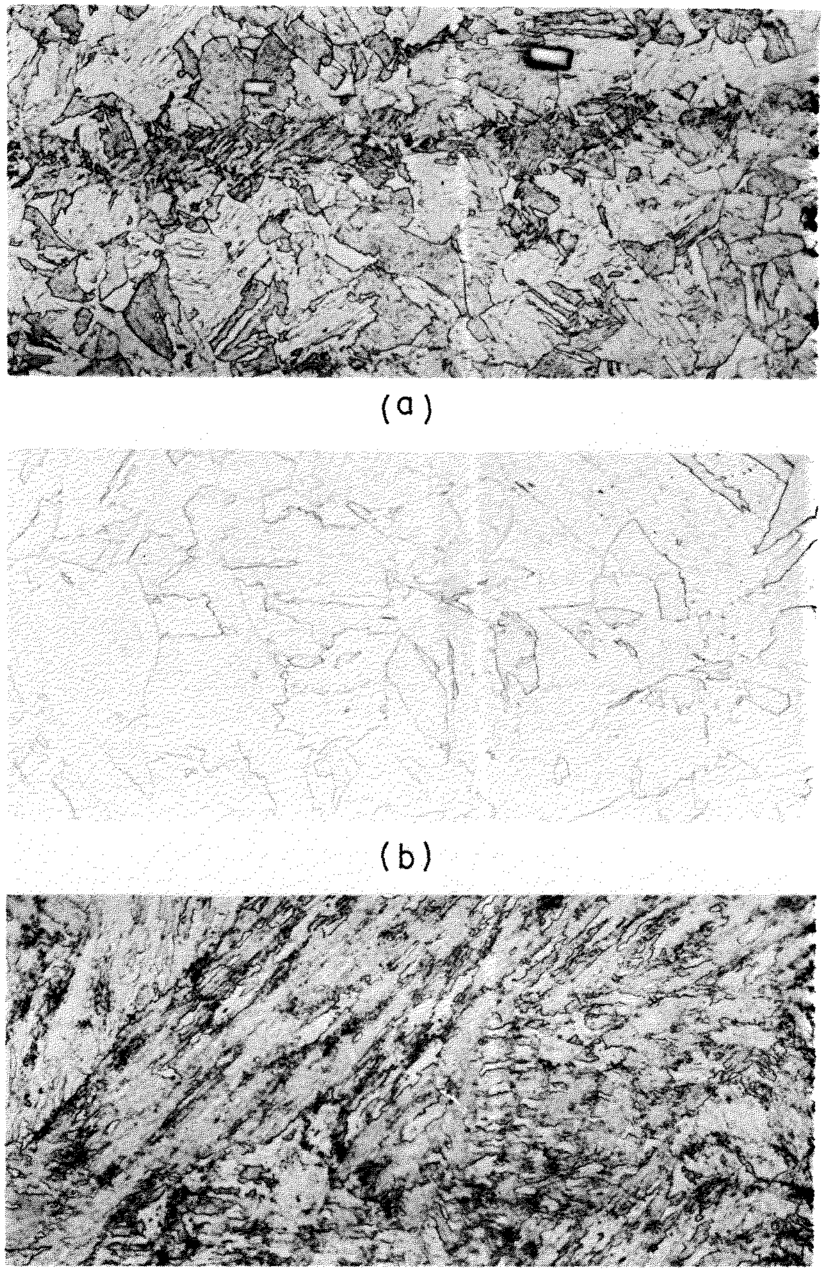
Specimen irradiations were conducted in the Materials Test Reactor (MTR) at the National Reactor Testing Station, Idaho; the Union Carbide Research Reactor (UCRR), Tuxedo, New York; the Low Intensity Test Reactor (LITR), Oak Ridge, Tennessee; and the Big Rock Point Power Reactor (BRPR), Charlevoix, Michigan. Specimen encapsulation techniques and experiment control equipment devised by NRL to provide desired exposure (thermal) environments have been described elsewhere (2,3).

An assumed fission spectrum distribution of neutrons indexed by iron-wire dosimetry formed the basis for the reported neutron fluence values. A fission, averaged cross section of 68 mb was selected for the $^{54}Fe(n,p)$ ^{54}Mn reaction. Methods for the conversion of fluence based on an assumed fission spectrum to fluence representing a calculated spectrum have been previously reported (4).

RESULTS

Experimental results for the individual plates and weld metals are listed in Table 3. It should be noted that the duration of the BRPR experiment (5) was about 9700 hr, or three to five times as long as equivalent fluence exposures in the LITR or UCRR. Table 3 lists the approximate irradiation time periods for each set of data.

Typical response to the low-temperature (<250°F) irradiation of 12-5-3 plate aged for 2 and 20 hr is shown in Fig. 2. Low ($\sim 5\times10^{18}$ n/cm^2) and high ($\sim 5\times10^{19}$ n/cm^2) fluence exposures are depicted in both graphs. The temperature shifts observed in the 30-ft-lb index level are similar to the <250°F irradiation response of A302-B steel (ASTM 6-in. reference plate, 65.5 ksi yield strength (6)). However, the higher strength



(a)

(b)

(c)

Fig. 1 - Composite microstructures of 12Ni-5Cr-3Mo maraging steel weldment representing (a) the base metal, (b) the heat-affected zone (HAZ), and (c) the weld metal (500X)

Table 3
Charpy-V Notch-Ductility Properties of 12-5-3 Maraging Steel Plates and Companion Weld Metals After Irradiation at Various Temperatures and Fluences

Material Code	Yield Strength (0.2% offset) (ksi)	Charpy-V* 30-ft-lb Transition Temperature (°F)										
		Initial Temp.	ΔT Increase Due to Irradiation									
			<250°F					550°F		585°F	650°F	740°F
			0.5† 200‡	0.6 300	0.7 200	5.5 1900	8.5 2400	3.0 1200	4.8 3300	7.3 9700	4.1 1900	5.6 1900
J1	179.1	-180	45	–	–	–	205	–	–	130	–	–
J3	170.6	-200	55	–	–	160	–	–	–	–	–	–
J4	162.8	-160	25	–	–	–	185	–	–	–	–	–
J5	176.2	-165	–	–	50	–	–	–	–	–	–	–
J6	184.4	-175	–	–	50	165	–	–	70	–	70	140
J7	181.5	-180	–	–	30	–	–	–	–	–	–	–
J8	162.6	-195	35	–	–	155	–	–	145	205	145	180
12-5-3 Weld	179.0	-70	–	≤20	–	115	–	110	–	–	–	–
17-2-3 Weld	178.8	-175	–	≤20	–	145	–	110	–	–	–	–

*Plate: Transverse to primary rolling direction.
Weld: Transverse to welding direction.
†Fluence (x 10^{19} n/cm^2 > 1 MeV).
‡Irradiation time (hr).

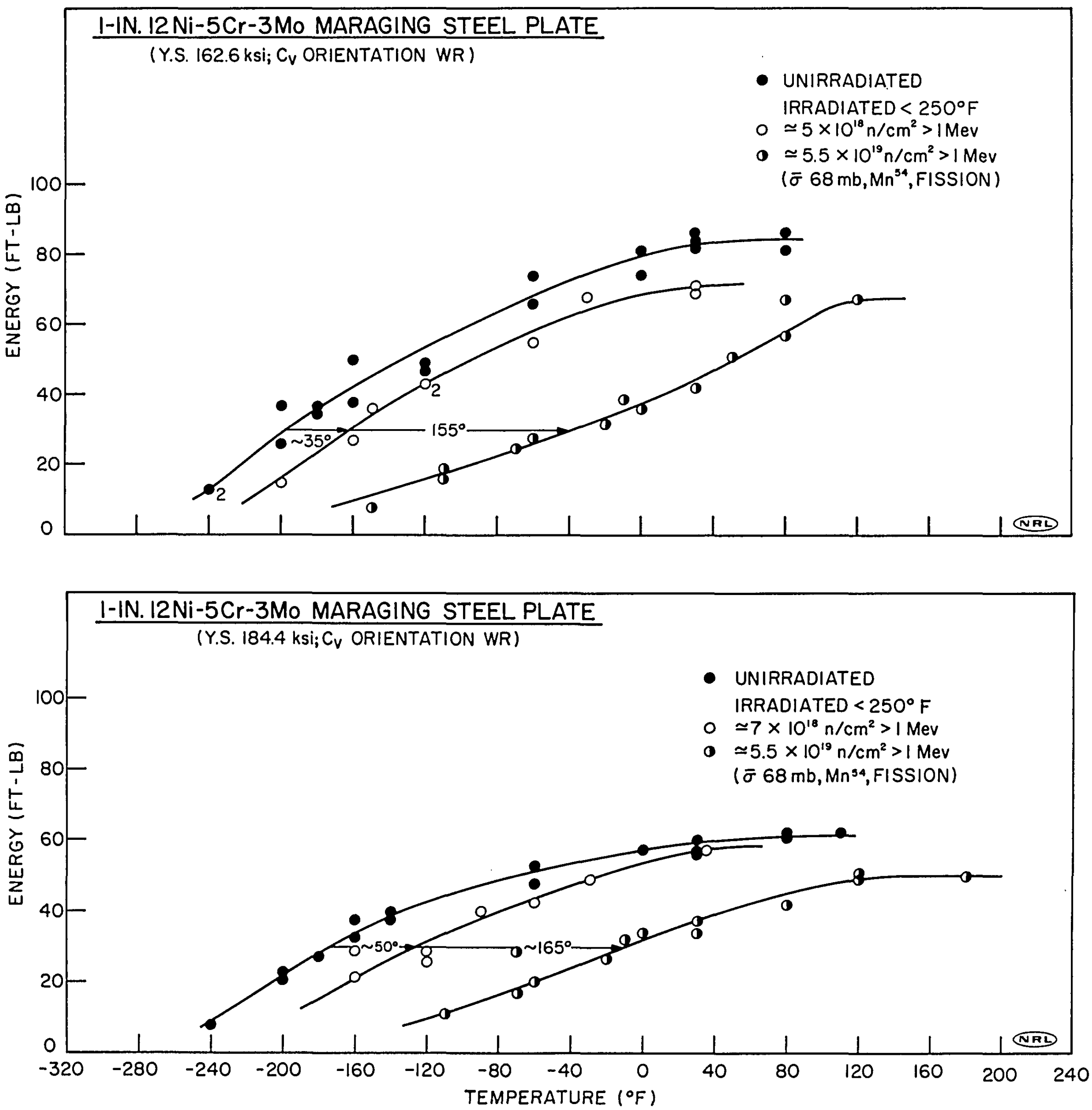


Fig. 2 - Charpy-V notch-ductility behavior of 12Ni-5Cr-3Mo maraging steel plates (160- and 185-ksi yield strength) before and after irradiation at <250°F to two levels of neutron fluence

12-5-3 alloy is distinctly superior in its low initial 30-ft-lb temperature (-200/-160 vs +15/30°F). The lowering of the full-shear energy shelf would appear to be the parameter of most concern, particularly for fluences greater than those reported in Fig. 2.

Figure 3 presents results for the same plate materials after irradiation at 585°F in the commercial power reactor (BRPR). Limited data on the postirradiation annealing response are also shown. Comparing the upper graphs of Figs. 2 and 3 (2-hr aged condition), the increase in the 30-ft-lb temperature following the 585°F BRPR exposure is of the same order of magnitude as that which could be projected for an equivalent fluence exposure at < 250°F. A marked decrease in the full-shear energy shelf is also seen. By contrast, data for 20-hr of aging (lower graphs) indicate a smaller 30-ft-lb temperature shift and a smaller reduction in the energy shelf. Further, the elevated temperature (585°F) irradiation response of the 20-hr aged condition is somewhat less than that of the A302-B reference plate (130 vs 165°F ΔT). However, postirradiation annealing at 700°F was not as effective in embrittlement relief of either the 2- or the 20-hr aged condition as has been observed for A302-B and other mild alloy steels.

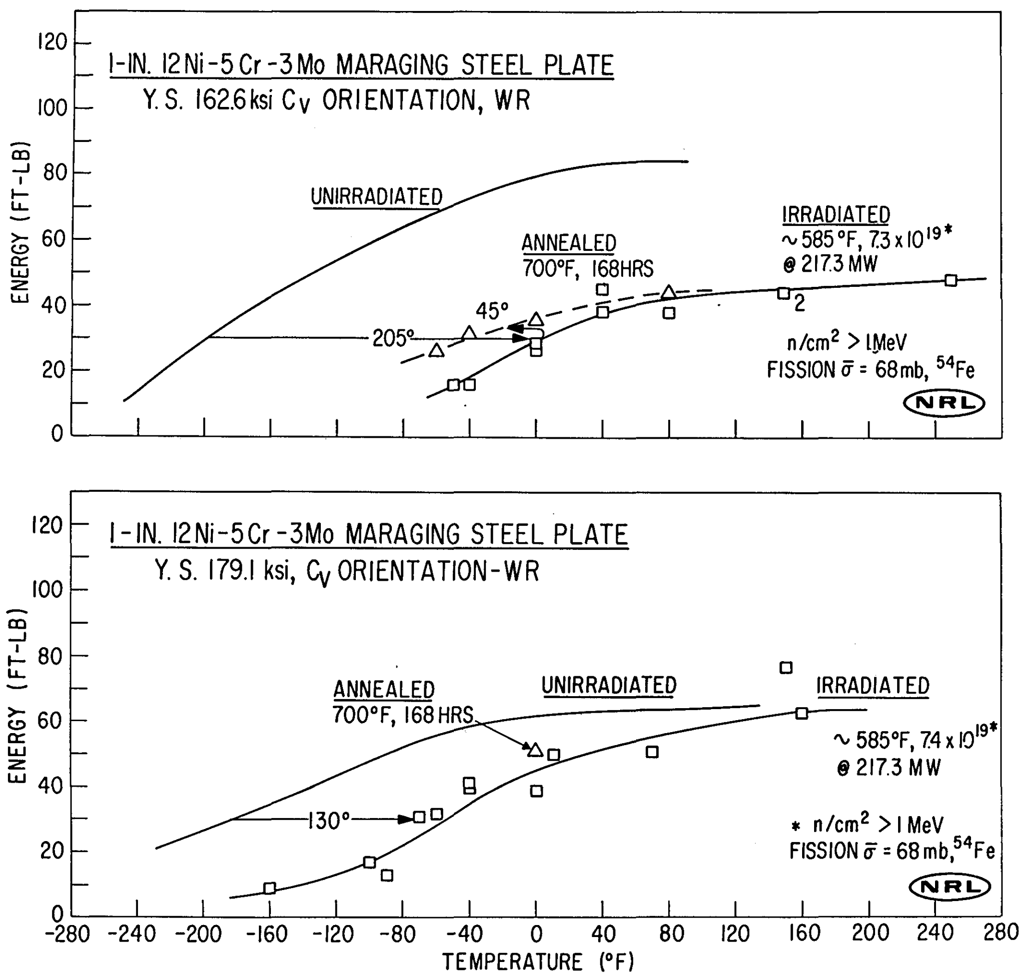


Fig. 3 - Charpy-V notch-ductility behavior of 12Ni-5Cr-3Mo maraging steel plates (160- and 185-ksi yield strength) before and after irradiation at 585°F in the Big Rock Point Reactor (a commercial power plant) to a fluence of 7.3 x 10^{19} n/cm^2 at a location of low neutron flux

The departure of 12-5-3 plate from anticipated embrittlement trends led to further testing of its irradiation response at elevated temperatures. The results of a series of experiments conducted at 550, 650, and 740°F are shown in Figs. 4 and 5. Unexpectedly, the notch-ductility behavior following 550°F irradiation (3300 hr) was the same as that following 650°F irradiation (1900 hr). Moreover, both 2- and 20-hr aged conditions experienced their greatest increases in 30-ft-lb temperature and most marked losses in shelf energy with 740°F irradiation (1900 hr). To correctly qualify this phenomenon, unirradiated specimens from both plates were thermally conditioned for time periods equal to the irradiation periods. The results are shown, superimposed on the irradiation data, in Figs. 4 and 5. In all cases, the extensive property changes resulting from thermal conditioning are indicated. Thus, progressive aging with thermal exposure at temperatures well below the initial maraging temperature (900°F) has been revealed. With one exception (2-hr aged condition, 550°F irradiation), the apparent continuation of the aging process more than accounts for the observed shifts of the 30-ft-lb index level with 550 to 740°F irradiation exposure. Figures 4 and 5 would indicate that the effects of low-temperature aging reach their maximum (saturate) in the time interval between 1900 and 3300 hr. However, studies of longer duration have revealed that thermal environments continue to degrade notch-impact properties with time in service. Limited results from specimens thermally conditioned at 585°F for approximately one year are shown in Figs. 4 (Plate J8, 160 ksi) and 6 (Plate J1, 180-ksi yield strength). It is noted that the property changes are more extensive than would be predicted from shorter duration (1900- and 3300-hr) experiments at comparable temperatures. The further elevation of the 30-ft-lb temperature and lowering of the full-shear energy shelf with time at temperature are highly significant to overall alloy stability at relatively low service temperatures.

Results obtained with thermally conditioned tensile specimens are in basic agreement with findings on thermally conditioned Charpy-V specimens. These results are shown, with data for low-temperature irradiations, in Table 4. Both the irradiation and the thermal conditioning treatments increased the yield and tensile strength of the plate materials without appreciably altering the ductility parameters.

The response of two weld metals to irradiation at less than 250°F and at 550°F is shown in Fig. 7. The general level of radiation embrittlement sensitivity observed with the various plates is also depicted by the welds. The initial shelf energy of the 12-5-3 weld is shown to be higher than that of the 17-2-3; however, the values for both welds are comparable after irradiation. The paucity of weldment stock precluded any thermal conditioning studies in this case.

DISCUSSION

A review of the data generated by this study indicates that shifts in the 30-ft-lb index temperature are about the same order of magnitude as those of other, lower strength, steels with equivalent (<250°F) exposure. Pressure-vessel steels, when evaluated with elevated temperature irradiation, normally demonstrate a partial annealing of neutron damage production. Thus, exposures at elevated temperatures are not as detrimental as at low temperatures (1). This was not found to be the case for 12-5-3 maraging steel, as illustrated in Fig. 2. The analysis suggests strong evidence of an aging problem. This opinion is well supported by the results of thermal conditioning treatments shown in Figs. 4, 5, and 6. Moreover, steel aged for 2 hr appears more unstable with respect to property changes with time and temperature than when initially aged for 20 hr.

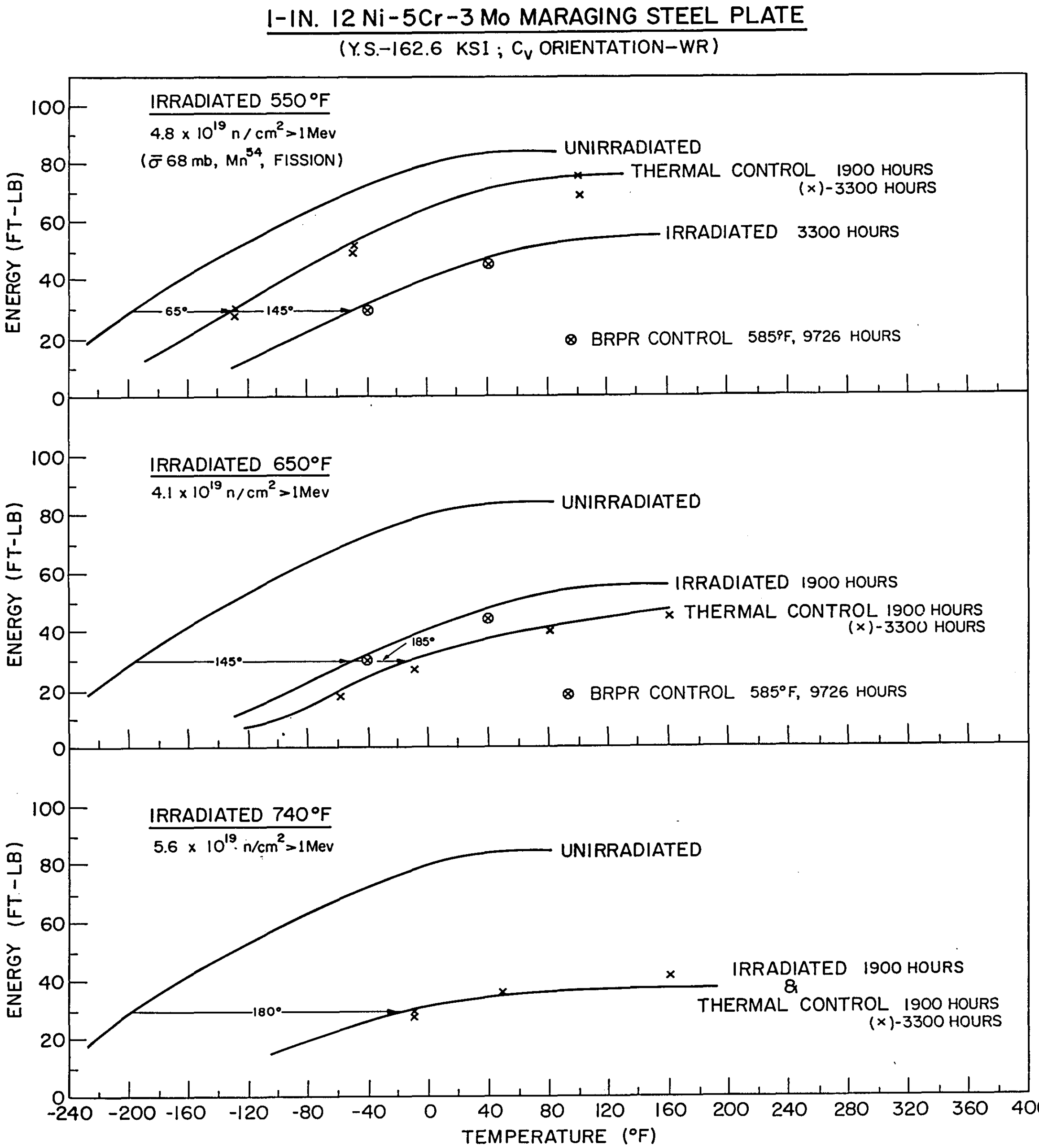


Fig. 4 - Charpy-V notch-ductility behavior of 12Ni-5Cr-3Mo maraging steel plate (160-ksi yield strength) before and after conditioning at 550, 650, and 740°F and after irradiation at the same temperatures. BRPR thermal control data representing long-term conditioning at 585°F are shown for comparison on the 550 and 650°F data plots.

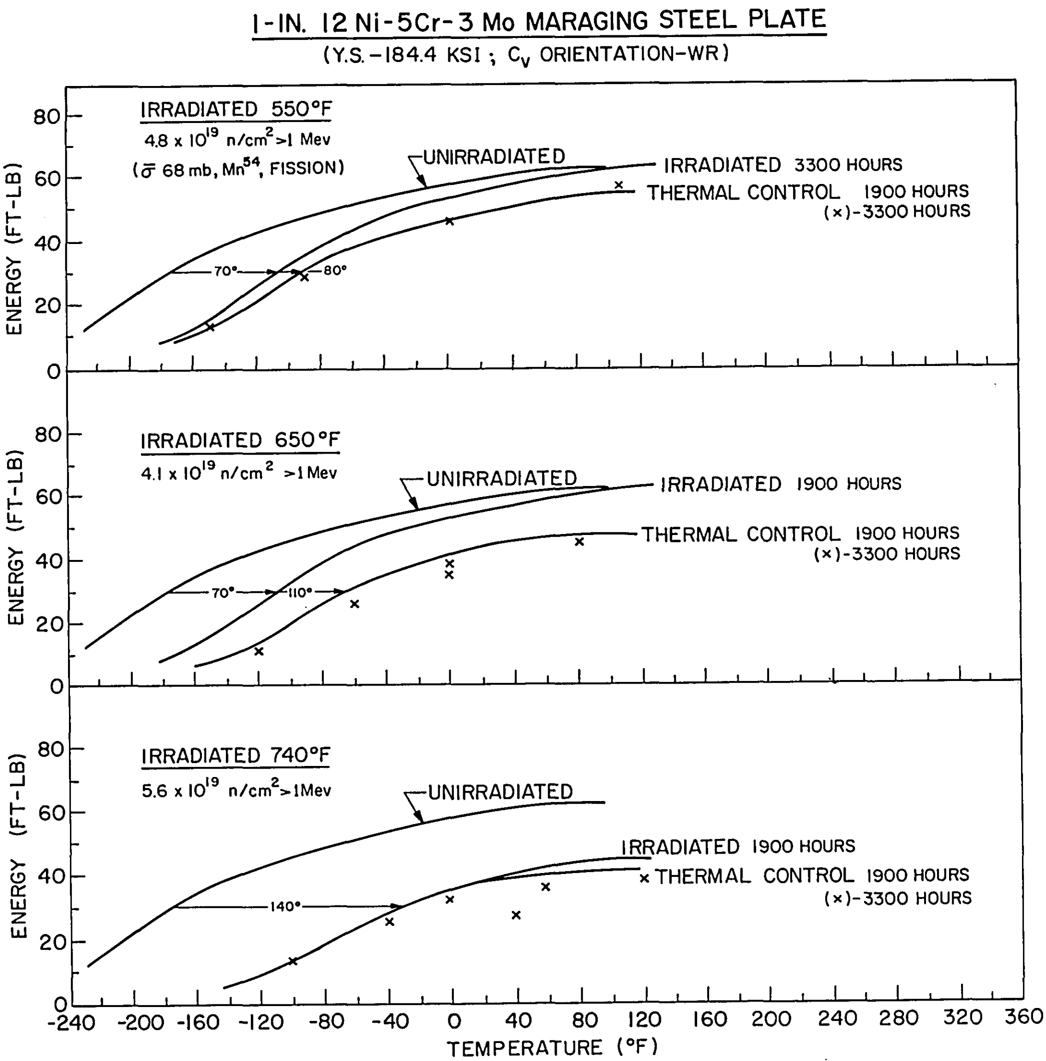


Fig. 5 - Charpy-V notch-ductility behavior of 12Ni-5Cr-3Mo maraging steel plate (185-ksi yield strength) before and after conditioning at 550, 650, and 740°F and after irradiation at the same temperatures.

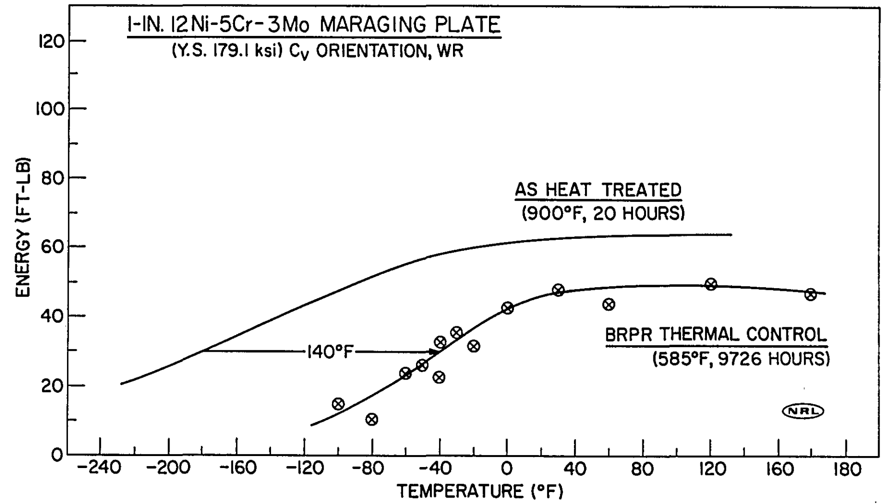


Fig. 6 - Charpy-V notch-ductility behavior of 12Ni-5Cr-3Mo steel plate (180-ksi yield strength) before and after conditioning at 585°F for approximately one year

Data developed on notch-ductility characteristics following low-temperature aging treatments are reinforced by tensile data listed in Table 4. The aging-induced changes in strength are noted to be greater than those measured for cold irradiations; however, the reduction of area and total elongation values do not indicate a significant deterioration in ductility. The overall, tensile properties are improved, and notch-impact parameters are adversely affected by extended service within the temperature range investigated.

A mismatched weld composition (17Ni-2Co-3Mo) is shown to be somewhat less sensitive to radiation than a matching weld metal in terms of the upper energy shelf depression (Fig. 7). The 17-2-3 weld also indicates an advantage of a lower initial 30-ft-lb temperature (-175 vs -70°F). However, the 30-ft-lb temperature shifts of both test welds were observed to be about equal. As with the plate materials, neither weld composition indicated any pronounced self-annealing at an elevated (550°F) irradiation temperature.

CONCLUSIONS

A series of low (<250°F) and elevated temperature (550 to 740°F) irradiation experiments has been conducted with 12Ni-5Cr-3Mo maraging steel. Interest in this type of steel for nuclear structural applications stems from attractive preirradiation strength and notch-ductility behavior. Fluence levels for plate and weld metals ranged from 0.5 to 7.3×10^{19} n/cm^2 >1 MeV. The Charpy-V 30-ft-lb transition temperature was selected as one arbitrary index for comparisons of irradiation performance. The following observations were drawn from this study.

1. The Charpy-V 30-ft-lb temperature behavior of 12-5-3 plate with 250°F irradiation is comparable to that of carbon and low-alloy pressure-vessel steels. Prior aging by heat treatment does not appear to be of major consequence in irradiation performance at this temperature range.

2. The thermally enhanced self-annealing of neutron-induced damage during 550 to 740°F radiation exposure, normally observed with lower strength steels, is partially or wholly masked in the 12-5-3 alloy by a competing aging process.

Table 4
Tensile Properties of 12-5-3 Maraging Steel Plates and Companion Weld Metals After Irradiation and With Long-Term Thermal Conditioning (Controls)

Material Code	Fluence ($\times 10^{19}$ n/cm^2 > 1 MeV)	Temp. (°F)	Time (hr)	Yield Strength* (0.2% offset) (ksi)	Tensile Strength† (ksi)	Elongation (%)	Reduction of Area (%)
J1	0	RT	—	179.0	185.1 A	15.5	64.0
	0.7	<250	200	193.6	197.0 B	13.6	64.1
	0.5	<250	200	195.2	195.2 B	13.2	62.9
	8.5	<250	2400	206.8	210.1 B	12.0	62.2
J3	0	RT	—	170.5	175.5 A	16.3	66.0
	0.5	<250	200	180.4	182.8 B	13.0	65.6
J4	0	RT	—	162.8	170.9 A	16.0	64.4
	0.5	<250	200	186.0	186.2 B	12.8	63.8
	8.5	<250	2400	195.9	201.0 B	6.6	40.4
J5	0	RT	—	176.2	185.3 A	13.5	58.3
	0	RT	—	176.8 L	184.4 A	16.0	61.1
	0.7	<250	200	193.6	196.0 B	12.5	65.3
J6	0	RT	—	184.4	190.0 A	13.5	55.7
	0	RT	—	181.6 L	187.4 A	15.0	62.2
	0.7	<250	200	195.3	188.3 B	12.8	65.0
	5.5	<250	1900	206.5	210.5 B	12.0	45.8
	0 (Control)	550	1900	188.8 L	195.9 B	15.5	66.9
	0 (Control)	740	1900	209.2 L	217.9 B	15.7	60.2
J7	0	RT	—	181.5	188.5 A	14.5	62.3
	0	RT	—	180.0 L	186.6 A	15.0	64.0
	0.7	<250	200	195.8	196.8 B	11.7	65.5
J8	0	RT	—	162.6	170.6 A	15.8	64.1
	0	RT	—	161.0 L	168.8 A	16.8	67.4
	0.5	<250	200	174.4	179.2 B	14.0	67.2
	0 (Control)	550	1900	170.7 L	180.3 B	17.0	68.2
	0 (Control)	740	1900	213.0 L	221.5 B	15.3	63.6
12-5-3 Weld	0	RT	—	179.0 X	188.0 B	16.0	62.5
	0.7	<250	200	189.7 X	195.3 B	15.3	43.4
	5.5	<250	1900	201.8 X	205.0 B	9.5	60.0
17-2-3 Weld	0	RT	—	178.8 X	187.5 B	16.0	64.7
	0.7	<250	200	194.0 X	195.2 B	13.2	43.4
	5.5	<250	1900	198.5 X	200.8 B	11.8	46.6

*Transverse (WR) specimens except as noted.
L — Longitudinal (RW) specimen.
X — All-weld specimen
†A — 0.505-in.-diam specimen.
B — 0.252-in.-diam specimen.

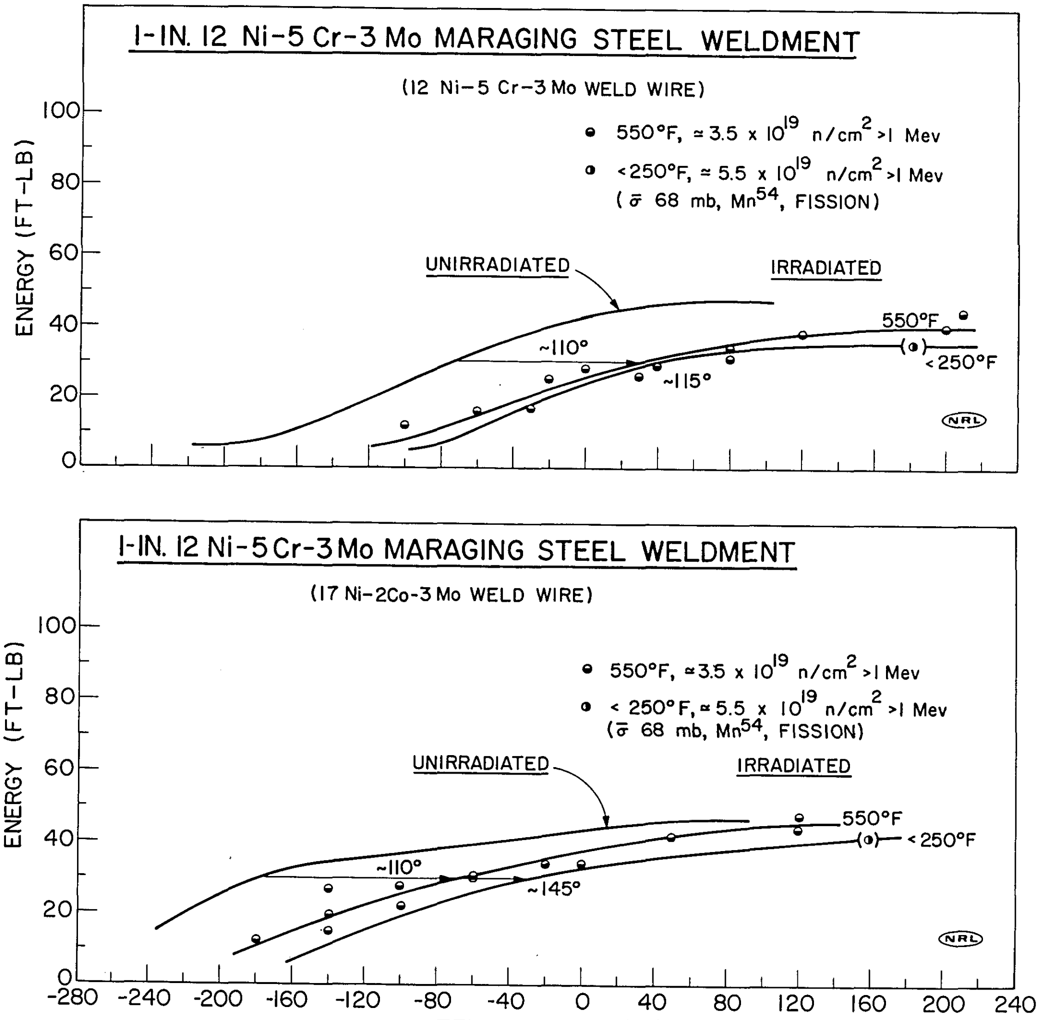


Fig. 7 - Charpy-V notch-ductility behavior of 12Ni-5Cr-3Mo and 17Ni-2Co-3Mo maraging steel weld metal deposits (180-ksi yield strength) before and after irradiation at <250°F and at 550°F

3. The TIG weld deposits of matching and mismatched (17Ni-2Co-3Mo) compositions parallel the performance characteristics of the 12-5-3 parent plate with $<250^\circ$F and 550°F radiation exposures.

4. The thermal environment of the 12-5-3 weldments during service is equally as important as the lifetime neutron fluence. Progressive aging may account for nearly all of the subsequent changes in notch ductility and strength.

5. Thermal exposures as low as 585°F extended for one year demonstrated a cumulative, adverse effect on notch ductility beyond projections from 1900- and 3300-hr data for comparable temperature conditions.

6. A marked increase in yield and tensile strength was observed with elevated temperature irradiation and independently with thermal conditioning. Only slight losses in the percentage reduction of area and elongation accompanied these strength changes.

7. The depression of the Charpy full-shear energy shelf to a value approaching 30-ft-lb with high-temperature, high-fluence exposures could pose a material limitation. Service above 650°F may cause losses in the Charpy energy shelf level, resulting in notch-sensitive material subject to low-energy, ductile-tear fracture.

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13. ABSTRACT

Changes of Charpy-V-notch ductility and tensile strength in neutron-irradiated 12Ni-5Cr-3Mo maraging steel have been evaluated following low (< 250°F) and elevated (550 to 740°F) temperature exposure. The study was performed with six heats of 1-in.-thick plate material aged at 900°F for 2 and 20 hr to nominal yield strengths of 160 and 180 ksi, respectively. The long-term thermal stability of both heat-treatment conditions was investigated for the conditions of irradiation. The < 250°F and 550°F irradiation performance of matching (15-5-3) and mismatching (17Ni-2Co-3Mo) TIG weld deposits maraged to 180 ksi yield strength was also assessed in this study.

Changes in the general properties of the 12-5-3 maraging steel plate and companion weld metals were found to be rather small with < 250°F exposures, indicating good resistance to neutron-induced embrittlement. However, a marked deterioration of notch-ductility properties with long-term exposure at elevated temperature was revealed and traced to a nonnuclear thermal instability. The observed instability is believed to be a continuation of aging processes at temperatures well below the initial maraging temperature. Extended time-at-temperature treatments indicate that service above 550°F may produce sufficient changes in properties for failure by low-energy tear. Aging treatments of 1900 to 3300 hours' duration increased the yield and tensile strengths of the 12-5-3 alloy by as much as 52.0 and 53.3 ksi, respectively, while not altering appreciably the percentage of elongation and the reduction in area.

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Structural steel						
Neutron embrittlement						
Neutron-embrittlement relief						